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DETERMINATION OF ANTIMONY IN  
ENVIRONMENTAL SAMPLES BY A  
SEMI-AUTOMATED HYDRIDE  
GENERATION-ATOMIC ABSORPTION  
METHOD

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Environment

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## ABSTRACT

Determination of antimony in environmental samples  
by a semi-automated hydride generation - atomic absorption method

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A semi-automated atomic absorption method for determining antimony in soils, sediments, vegetation, water and air-borne dust is presented. Samples and standards are digested with a mixture of sulfuric, nitric and/or perchloric acids in calibrated test tubes. The digestates are made up to 15 ml with distilled water and processed through an automated analytical system.

Antimony is converted to a gaseous hydride which is atomized by passage through a heated quartz tube furnace. The atomic absorption signal is recorded at 217.6 nm. A Technicon pump and sampler are coupled with an atomic absorption spectrophotometer to achieve production of steady state absorption signals. Digestates can be analysed at a rate of 20 per hour.

The method is relatively free of interferences by commonly occurring elements. Tellurium may interfere at concentrations greater than 1  $\mu\text{g/ml}$ . The relative standard deviation of the method is 8.5% at antimony concentrations ranging between 0.15 and 5.0  $\mu\text{g/g}$ . The precision of the determinative step for 15 ng Sb/ml is better than 2.5% RSD. The sensitivity and detection limit of the method are 0.44 ng/ml and 0.26 ng/ml respectively. Accuracy is established by replicate and analyses of NBS Orchard Leaves and Pine Needles samples as well as parallel analysis of different sample types by the Rhodamine B method. The mean recovery of added antimony in lake and rain water samples is 97.4%.

Ambient air concentrations for different Ontario locations are also presented.



## INTRODUCTION

The amount of antimony used in industrial processes in the USA has shown a significant increase over the past decade<sup>1</sup>. Similar increases are expected to have occurred in Canada. Environmental scientists and engineers require data on antimony in order to evaluate possible problems associated with the steady increase in the antimony burden of the overall North American environment.

The past data on metals in ambient air of many cities in the United States was obtained by USEPA\* using an emission spectrographic method for multi-element analyses of airborne dust trapped on glass fibre filters<sup>2</sup>. This method is quite insensitive for antimony and the results obtained, if any, are not reliable when antimony is one of the several elements determined simultaneously. Even if handled in a special way, the method is subject to the interfering effect of numerous parameters. Concentrations of antimony in a great majority of environmental samples are below 5 ppm; lake, rain and drinking waters invariably contain less than 0.1 ppm antimony. It is necessary, therefore, that the analytical method should be capable of accurately measuring 5 ng/ml antimony in prepared solutions of these samples. The procedure should also involve fewer steps and require smaller quantities of sample and reagents than existing methodologies. The conventional flame atomic absorption spectrophotometric methods do not have the required sensitivity. The Rhodamine B colorimetric method<sup>3</sup>, although sufficiently sensitive, is tedious and requires a large amount of sample for analysis. The neutron activation technique although suitable, is complex and costly, and often involves complicated wet chemistry for prior separation of antimony from sample matrices.

The conventional flameless graphite furnace atomic absorption technique<sup>4</sup>, while providing many excellent advantages, is subject to non-specific interferences from light scattering

\* United States Environmental Protection Agency

and broadband molecular absorption by the sample matrix. These interferences cause additional absorption not produced by the element of interest and give erroneous analytical results. Volatility losses during the ashing cycle may be possible.

Akira et al<sup>5</sup>, following the work of others who employed dc discharge or microwave induced plasma as the measurement technique, have used dc plasma for determining antimony in waste water and sea water samples. Their method is based on the separation of antimony as the hydride in a liquid nitrogen trap and does not offer any special advantage over the hydride atomic absorption spectrophotometric approach used in this paper.

Vijan et al<sup>6-10</sup> have published methods for the determination of arsenic, selenium, lead and tin in environmental samples by automated generation of volatile hydrides of these elements, followed by their atomization in a heated quartz tube and measurement of their atomic absorption signals. These methods have become very popular; many papers have been published on the subject and are discussed in the above references. Inhat and Miller<sup>11</sup> have reviewed the different hydride generation systems and atomization techniques.

Pierce and Brown<sup>4</sup> have demonstrated the superiority of automated hydride generation over manual and graphite furnace atomization methods. Fiorino et al<sup>12</sup> determined antimony in food samples, but their absorption signals were transient as opposed to the steady-state signals given by the continuous automated generation of stibine. Chan and Vijan<sup>13</sup> have recently published a method, similar to one being presented here, for the determination of antimony in rocks.

## EXPERIMENTAL

### Reagents

Perchloric acid (ACS 70-72%), nitric acid (ACS 70-71%), and sulfuric acid (ACS 95.5-96.5%) were used. All other chemicals used were ACS analytical-reagent grade.

*Borohydride solution.* Dissolve 3.0 g of sodium borohydride (Fisher Scientific) and 0.3 g of sodium hydroxide in 300 ml of distilled water. Use fresh. Solutions may be stored at 5°C. for a few days.

*Stock standard antimony solution.* 1000 mg l<sup>-1</sup> commercial standard from SbCl<sub>3</sub>). Prepare working standard solutions (5-30 ng Sb ml<sup>-1</sup>) by serial dilution of the stock standard with 10% sulfuric acid.

*Potassium iodide solution.* Dissolve 10 g of potassium iodide and 0.1 g of sodium hydroxide in 100 ml of distilled water.

### Apparatus

An atomic absorption spectrometer capable of giving a stable signal at the Sb 217.6-nm line is required. A Techtron Model AA5 equipped with HTV R106 photomultiplier tube and a 1-10 mV variable range strip chart recorder was used. The sampler and proportioning pump were Technicon modules. The quartz tube, 10 cm long and 0.6 cm i.d. with a 17 cm long inlet tube (0.3 i.d.) at the centre, was wound with 22-gauge asbestos-coated "Chromel A" heating wire (Sargent Welch, Cat. No. 585126). The cell was further insulated with a layer of asbestos tape and a layer of asbestos cord. The temperature of this furnace was controlled with a variable transformer. The argon flow was regulated by a calibrated flow-meter. A 4-place Sartorius analytical balance was used for weighing the samples. The flow system is shown in Fig. 1 and the optimal instrumental parameters are as

follows: wavelength, 217.6 nm; lamp current, 10 mA; slit width, 100  $\mu$ m; flow rate (argon), 300 ml min<sup>-1</sup>; instrument damping, D (maximum); recorder span, 2 mV full-scale; recorder speed, 0.25 cm min<sup>-1</sup>; atomizer temperature, 850  $\pm$  10°C ; sample time, 1 min; wash time, 2 min.

#### Sample Preparation

##### (a) Vegetation, Soils and Sediments:

About 100 mg of each sample was weighed accurately and transferred to an 18 x 150 mm test tube with a calibration mark at 15 ml. The test tubes were held in a 40-hole aluminum heating block. A blank and four standards representing 5, 10, 15 and 20 ng ml<sup>-1</sup> antimony were included in each set of samples by adding 0, 0.05, 0.10, 0.15 and 0.20 ml of 1.5  $\mu$ g ml<sup>-1</sup> solution of antimony to five separate test tubes using a 0.2 ml class A measuring pipet with 0.001 ml subdivisions. Five ml of a 3:6:1 mixture of concentrated sulfuric, nitric and perchloric acids were added to each test tube. The loaded aluminum block was heated to about 100°C on a hot plate. After the initial reaction subsided, the temperature was raised to about 140°C and held there until the appearance of white fumes (approximately 3 hours). The aluminum block was cooled to room temperature. Five ml of distilled water was added to each test tube. The contents were stirred and allowed to cool to room temperature. Each test tube was then filled to the 15 ml mark with distilled water and mixed. The insoluble material was allowed to settle and the clear supernatant transferred to the sample cups of the automatic sampler for determination of antimony.

(b) Hi-Vol Glass Fibre Filters:

Aliquots were cut out from each sample filter using #11 cork borer (5.2 sq cm) and digested with 2.5 ml of a 2:1 mixture of concentrated sulfuric and nitric acids in a manner similar to section (a) above. Reagent and filter blanks and a set of standards were carried through the entire decomposition procedure along with the samples.

(c) Waters:

Appropriate aliquots were mixed with 2.5 ml of the 2:1 sulfuric, nitric acid mixture in test tubes as in (a) and (b). The samples were evaporated to 2.5 ml on a hot plate, aided by an overhead infra-red heat lamp. The tubes were further heated until fumes of sulfur trioxide appeared. The samples were cooled and diluted to 15 ml with distilled water. A blank and standards were included with a set of samples and taken through the decomposition procedure.

Instrumental Procedure

The instrumental parameters, previously listed, were established and the equipment was allowed to warm up for 20-30 minutes. The quartz tube furnace, fastened securely to the top of a burner head, was aligned with the light beam to allow maximum light from the hollow-cathode lamp to reach the detector. The quartz tube was heated to the optimum temperature. The reagent lines were inserted into the appropriate solutions and the proportioning pump started. The argon gas was turned on immediately and the flow adjusted to  $300 \text{ ml min}^{-1}$ . The performance of the system was checked by the response of a control standard. The sample standards and reagent blank solutions were transferred to the sample cups held in the sampling tray. Every 10th cup was filled with a  $10 \text{ ng ml}^{-1}$  standard solution to monitor instrumental drift. The peak heights



of the standards were measured, a calibration graph drawn and the concentrations of antimony in the sample solutions were read from this graph.

## RESULTS AND DISCUSSION

### Interferences:

Under the experimental conditions of this method, the elements most likely to interfere are those whose ions react with sodium borohydride in strongly acidic media, either to form volatile hydrides, or to precipitate as the metal.

The interference may result from the alteration of the rate of reaction of co-precipitation of the analyte or both. The elements which form volatile hydrides are those of groups IVA, VA and VIA and those which precipitate as metals belong to groups IB, IIB and VIII.

Table I shows that the method is free of interference from large concentrations of the major matrix constituents such as aluminum, iron, calcium, magnesium, sodium, potassium and titanium. The concentrations of these elements in the prepared solutions of most samples are expected to be less than these values.

The stock arsenic and tellurium solutions used in the interference study contained traces of antimony as an impurity. A  $3 \mu\text{g ml}^{-1}$  tellurium solution produced an antimony signal equivalent to  $3 \text{ ng Sb ml}^{-1}$ . Progressively smaller peaks were obtained for 2 and  $1 \mu\text{g ml}^{-1}$  tellurium solutions. The possibility of molecular absorption was excluded by the use of the non-absorbing 217.0-nm line which produced no signals. A similar antimony signal was observed for  $10 \mu\text{g As ml}^{-1}$  solution. Also a 15% depression of the 10 ppb antimony signal is caused by  $10 \mu\text{g As ml}^{-1}$  or  $3 \mu\text{g Te ml}^{-1}$  solutions. Occurrence of arsenic and tellurium at these concentrations in the prepared solutions of samples is highly unlikely.

Common anions such as chloride, nitrate, perchlorate and phosphate do not interfere. Sulfite and sulfide ions interfere by producing a non-specific absorption peak due to hydrogen sulfide. However, both sulfites and sulphides are destroyed during the sample digestion and do not pose any problem.

#### Effect of Acid Concentration:

The acid concentration of the sample solution has an important effect on the degree of interference. With the potassium iodide line eliminated (Fig. 1), when the concentration of sulfuric acid in the sample was reduced from 10% to 1% (v/v), less than  $1 \mu\text{g ml}^{-1}$  of copper, selenium and tin began to cause significant depression of the 10-ppb antimony signal. Sets of 20 ng Sb  $\text{ml}^{-1}$  standards prepared in hydrochloric, nitric, perchloric and sulfuric acids of incremental concentrations between 0.1% and 30.0% (v/v) were processed twice through the analysis system (Fig. 1), once with potassium iodide reagent and a second time with distilled water pumping through channel 2. The results of this study are depicted in Fig. 2. The maximum absorbance of 20 ng Sb(V)  $\text{ml}^{-1}$  is 0.09 in the absence of potassium iodide versus 0.180 in its presence. This is an effect of the oxidation state of antimony and is explored later.

It is also evident from curve A that potassium iodide is ineffective as a reducing agent in a medium less than 2% acid. Its full potential is realized at acid concentrations greater than 7%. The other acids investigated behaved similarly, as did mixtures of acids.

Sulfuric acid is considered very suitable for solubilizing antimony in most chemical forms. The lower boiling acids such as nitric, hydrochloric, hydrofluoric and perchloric are easily expelled on heating to fumes of sulfur trioxide, thereby allowing close control of acidity of the prepared sample solutions. The use of nitric acid may be necessary if the samples contain organic matter.

Antimony halides are reported<sup>12</sup> to be volatile under certain conditions of digestion and the use of hydrochloric acid should be avoided during the digestion phase. However, no losses of antimony were discovered when up to 1 ml of hydrochloric acid was deliberately added to the samples and standards prior to digestion.

#### Effect of Oxidation State of Antimony:

Most digestion procedures are oxidative and convert antimony to the pentavalent state. Therefore, test solutions of antimony(V) were prepared by oxidizing a standard antimony(III) solution with 0.1 N potassium permanganate solution or from a 100 ppm stock solution prepared from antimony pentoxide.

In the absence of potassium iodide, a  $20 \text{ ng ml}^{-1}$  solution of antimony(III) produced a signal six times higher than that of antimony(V). When potassium iodide was present, antimony in either oxidation state produced signals of equal magnitude. The potassium iodide, therefore functions primarily as a reductant for antimony(V).

All standard antimony solutions prepared in 10% hydrochloric or sulfuric acids or their mixtures were found to be stable, except that some conversion of antimony(III) to antimony(V) occurs on storage. This was indicated by somewhat lower than the expected absorption peak heights obtained in the absence of potassium iodide. Lower acid concentrations appear to favour this conversion. However, this is of no concern since potassium iodide (10% w/v solution) is a component of the analytical train. It converts antimony(V) to antimony(III) prior to the determinative step and also increased the tolerance of the method to selenium by reducing it to the element. These findings are in agreement with those of Fiorini et al<sup>12</sup>. Potassium iodide solution concentrations varying between 5 to 10% w/v, (equivalent to 0.2 - 0.4% in the final mixed stream) were found to be adequate. A 10% solution was used

to allow for the reduction of other species such as iron(III). Other reducing agents such as chromium(II)<sup>13</sup> and sodium sulfite were investigated without success.

Detection Limit, Sensitivity, Precision and Accuracy:

The detection limit of the method is  $0.26 \text{ ng Sb ml}^{-1}$  or  $0.04 \text{ } \mu\text{g/g}$  on the basis of 100 mg sample weight. Ten recorder tracings obtained on an actual sample were used for calculating the detection limit of the method, defined as the concentration in  $\text{ng ml}^{-1}$  which corresponds to twice the standard deviation of the measurements taken near zero concentration.

The sensitivity and the linearity are illustrated in figure 2, together with some typical recorder tracings for actual sample solutions. The analytical calibration curve was found to be linear to about  $100 \text{ ng Sb ml}^{-1}$  under optimum conditions of instrument performance. The sensitivity i.e. concentration of Sb corresponding to an absorbance of 0.0044 (Fig. 2) is  $0.44 \text{ ng ml}^{-1}$ . Reagent blank peak heights varied from 0 to 1.5 chart divisions under normal operating conditions.

Ten replicate measurements of a  $15 \text{ ng ml}^{-1}$  antimony solution showed a R.S.D. of 2.5%. Table 2 contains the results of replicate analysis of NBS Orchard Leaves<sup>a</sup> and Pine Needles samples.

The precision of the overall determination for Orchard Leaves is 8.6% R.S.D. for samples weighing between 110 and 522 mg and 14% R.S.D. when 9 mg to 67 mg sample weights are used for the test. The weighing error alone can account for the decrease in precision in the latter case, yet the results are of acceptable accuracy. Excellent agreement with the NBS certified value of  $2.9 \pm 0.3 \text{ } \mu\text{g/g}$  is shown.

In the case of NBS Pine Needles, a mean antimony concentration of  $0.18 \text{ } \mu\text{g g}^{-1}$  agreed closely with the reported

uncertified value of  $0.20 \mu\text{g g}^{-1}$ . The precision was comparable with that of the Orchard Leaves. Similar precision data for a Hi-Vol filter sample is shown in Table 3. Table 4 contains the results obtained on 10 typical samples of different species of unwashed (NW) vegetation samples analysed by the present method and the Rhodamine B colorimetric method. The agreement is satisfactory.

Table 5 lists some typical antimony concentrations found in the ambient air of 41 Ontario locations. These results are based on the analysis of suspended particulate matter trapped on conventional Hi-Vol glass fibre filters<sup>6</sup>. A few stations showing higher concentrations of antimony are located near battery crushing and lead smelting plants.

Since arsenic is chemically related to antimony, it was determined in the same sample digestate. The ratio of arsenic to antimony varied between 0.1 and 20.0 (Table 6). However, the ratio showed a reasonable degree of constancy for a given station. It is seen that samples collected on different days show some variation in the antimony to arsenic ratios. This may be due to the changes in meteorological conditions and/or the contributions by polluting industries. Similar arsenic to antimony ratios were noted in the case of vegetation samples.

Three samples of soil and two of sediments were analysed several times by the present method and once by the Rhodamine B colorimetric method. The results are summarized in Table 7. The agreement between the two methods is good. The precision is comparable to that for vegetation and Hi-Vol results.

Table 8 contains the results obtained on lake and rainwater samples spiked with antimony. These water samples were sent in by a MOE regional laboratory as "unknown" samples.

A mean recovery of 97.4% was obtained. The data show that 0.2 nanogram antimony per ml can be measured with reasonable accuracy in water samples without any pre-concentration.

General Comments and Suggestions:

The analytical system described is very rugged. It has given continuous service without breakdown of any component for more than a year, except for occasional changes of worn manifold tubes. The baseline is so stable that low instrument damping may be used although, the use of a different instrument may change the baseline characteristics as well as the linear range of the calibration curve. The use of organic solvents or detergents, poisons the system and must be avoided. Accumulation of heavy metals in the reagent lines causes a drastic decrease in sensitivity. Pumping 1% (v/v) hydrogen peroxide, distilled water, 4% (w/v) ammoniacal EDTA, distilled water; in that sequence, through all the reagent lines usually cleans the manifold tubes and restores the sensitivity. If this treatment fails, the tubes should be replaced. It is important to disengage the roller head of the peristaltic pump and unstretch the manifold tubes at the end of the analysis. The concentrated sulfuric acid in the impinger drier requires replacement when the liquid level reaches to within 2-3 mm of the tip of the inlet tube.

It is important to turn on the carrier gas stream before or soon after the peristaltic pump is started. Failure to do so may cause fouling of the flow-meter and may also cause a minor flashback in the impinger because of back-firing of hydrogen. The temperature of the quartz tube may be varied within  $\pm 10^{\circ}\text{C}$  without significant effect on sensitivity but larger temperature fluctuations must be avoided. Air flow about the tube furnace should be just sufficient to remove evolved gases. Larger flows cause signal instability. The effect of the flow rate of carrier

gas on the antimony signal is significant and an accurate flow regulation is necessary for good precision. The use of nitrogen as a sparging gas causes a 30-40% loss in sensitivity compared with argon.

The use of an automatic sampler is not mandatory. The sample solutions can be fed to the sample channel manually and held there until the recorder pen begins to rise. The choice of 15 ml as the final volume of the prepared solution provides sufficient liquid for duplicate measurements. Any other convenient volume may be used if so desired.

The system can also measure arsenic with sensitivity and detection limit comparable to antimony in the same prepared solution.

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33-43



TABLE 1

Concentrations of elements  
which do not interfere<sup>a</sup>

Element	$\mu\text{g ml}^{-1}$	Equivalent conc. <sup>b</sup> in solid samples
Ca	4000	60%
Mg	4000	60%
K	4000	60%
Na	4000	60%
Al	4000	60%
Fe	4000	60%
Zn	200	3%
Pb	200	3%
Ti	200	3%
Cd	200	3%
Cu	20	3000 ppm
Ni	10	1500 ppm
Co	40	6000 ppm
Bi	10	1500 ppm
As	5	750 ppm
Se	20	3000 ppm
Sn	20	3000 ppm
Te	1	150 ppm

a 10 ng Sb ml<sup>-1</sup> solutions were used for interference study

b Based on 0.1g sample in 15 ml of solution

TABLE 2

DETERMINATION OF ANTIMONY IN  
NBS ORCHARD LEAVES<sup>a</sup> (1571) AND PINE NEEDLES (1575)

Sample	Sample wt. (mg per 15 ml)	ng Sb ml <sup>-1</sup>	Dilution	Ug Sb g <sup>-1</sup>	Statistic
Orchard Leaves variable weights  - High -	521.6	10.4	10x	3.00	Mean = 3.02 S.D. = 0.26 R.S.D. = 8.5
	348.8	6.97	10x	3.00	
	284.6	12.2	5x	3.23	
	253.0	11.5	5x	3.45	
	226.6	9.00	5x	2.98	
	178.2	6.75	5x	2.85	
	170.0	6.24	5x	2.70	
	164.7	5.98	5x	2.70	
	128.4	4.81	5x	2.85	
	109.8	4.94	5x	3.30	
- Low -	9.2	1.90	1x	3.10	Mean = 3.00 S.D. = 0.42 R.S.D. = 13.
	13.8	3.10	1x	3.37	
	18.6	3.95	1x	3.18	
	19.4	3.70	1x	2.67	
	35.5	6.30	1x	2.66	
	35.7	6.00	1x	2.52	
	43.3	8.80	1x	3.05	
	49.4	12.4	1x	3.76	
	60.2	13.0	1x	3.24	
	67.0	11.0	1x	2.46	
Pine Needles	150.0	2.04	1x	0.20	Mean = 0.18 S.D. = 0.014 R.S.D. = 7.8
	150.0	1.82	1x	0.18	
	150.0	1.70	1x	0.17	
	150.0	2.04	1x	0.20	
	150.0	1.70	1x	0.17	
	150.0	1.70	1x	0.17	
	150.0	1.70	1x	0.17	
	150.0	1.93	1x	0.19	
	150.0	1.59	1x	0.16	
	150.0	1.93	1x	0.19	

a - NBS certified value is  $2.9 \pm 0.3$  ug/g

b - NBS uncertified value is 0.2 ug/g

TABLE 3

PRECISION OF ANTIMONY DETERMINATIONS  
IN A HI-VOL FILTER SAMPLE<sup>a</sup>  
(2.84 sq. cm. aliquot per 15 ml)

Antimony µg/filter	Mean	S.D.	R.S.D %
44.2			
41.7			
41.7			
36.7	40.0	3.17	7.93
34.2			
36.3			
41.7			
40.4			
41.7			
41.7			

a - circles punched out from different areas of the same filter

TABLE 4

DETERMINATION OF ANTIMONY IN GROUND VEGETATION SAMPLES

Sample No. 1976	Antimony $\mu\text{g/g}$	
	Present <sup>a</sup> Method	Rhodamine B <sup>b</sup> Method
1224 NW	7.5	7.3
1231 NW	3.2	3.2
8059 NW	4.3	4.4
8063 NW	3.8	3.9
8067 NW	32	30
8068 NW	33	35
9351 NW	0.39	0.5
9356 NW	0.17	0.2
9357 NW	0.26	0.2
9359 NW	0.17	0.2

a - Mean of triplicate measurements

b - Mean of duplicate measurements

TABLE 5

DETERMINATION OF ANTIMONY IN AMBIENT AIR  
(RESULTS BASED ON THE ANALYSIS OF HI-VOL FILTERS)

STATION #	DATE OF COLLECTION	ng Sb/m <sup>3</sup>	STATION #	DATE OF COLLECTION	ng Sb/m <sup>3</sup>
15006	06/06/77	0.39	44010	31/05/77	4.44
15007	06/06/77	0.37	46025	25/05/77	1.29
22031	31/05/77	1.24	46041	29/06/77	731.8
22032	31/05/77	1.27	46045	07/08/77	10.0
22056	19/04/77	3.31	46046	07/08/77	8.58
22069	19/04/77	3.84	56076	18/06/77	1.71
27030	05/05/77	9.56	56077	24/06/77	1.40
27031	06/06/77	0.75	71001	18/05/77	0.59
29011	06/07/77	2.92	71022	13/05/77	1.17
31001	10/06/77	1.20	71042	19/04/77	6.52
31018	02/07/77	2.23	71049	13/05/77	0.91
31057	20/07/77	10.3	72014	30/05/77	0.65
31058	02/07/77	22.5	75020	12/05/77	0.04
31064	30/06/77	45.1	75021	05/06/77	1.09
31065	02/07/77	3.99	77016	13/05/77	0.05
33002	16/05/77	3.88	77026	08/03/77	0.29
34007	13/05/77	2.66	77028	24/02/77	0.23
35003	27/06/77	2.28	77069	15/10/76	0.23
35005	27/06/77	0.97	77070	03/05/77	1.07
35033	22/05/77	1.43	77076	13/05/77	0.05
35037	16/04/77	1.20			

TABLE 6

RATIO OF ARSENIC TO ANTIMONY IN HI-VOL FILTERS  
FROM AN INDUSTRIAL STATION (46041)

Date Exposed	ng Sb/m <sup>3</sup>	ng As/m <sup>3</sup>	As/ Sb
02/07/76	875	557	0.64
29/06/76	304	238	0.78
08/06/76	88	65	0.74
11/05/76	1229	511	0.42
12/04/76	351	180	0.51
08/03/76	732	710	0.97
Mean =			0.68
Mean Dev. =			<u>+0.15</u>

TABLE 7

DETERMINATION OF ANTIMONY IN SOILS AND SEDIMENTS

Sample	No. of Detmns.	Mean $\mu\text{g Sb g}^{-1}$ present method	R.S.D. %	$\mu\text{g/g}$ Rhodamine B Method
Soil -1	10	0.32	14.2	0.41
Soil -2	8	1.13	8.42	1.02
Soil -4	11	1.81	6.87	1.95
Sediment -1	10	4.85	6.50	4.63
Sediment -2	10	2.73	7.00	2.70

TABLE 8

## DETERMINATION OF ANTIMONY IN WATER SAMPLES

Sample	ng Sb ml <sup>-1</sup> found in sample	ng Sb ml <sup>-1</sup> added <sup>a</sup>	ng ml <sup>-1</sup> found in sample & spike	% Recov.
Lake Water #1	<0.2	20	18.9	94.5
2	<0.2	20	19.4	97.0
3	<0.2	15	16.1	107.3
4	<0.2	15	15.0	100.0
5	6.1	20	25.5	97.7
Rain Water #1	1.2	-	-	
2	0.5	-	-	
3	<0.2	-	-	
4	1.7	-	-	
5	<0.2	4.0	3.6	90.0
6	<0.2	40	42	95.2
Mean % Recov.				97.4



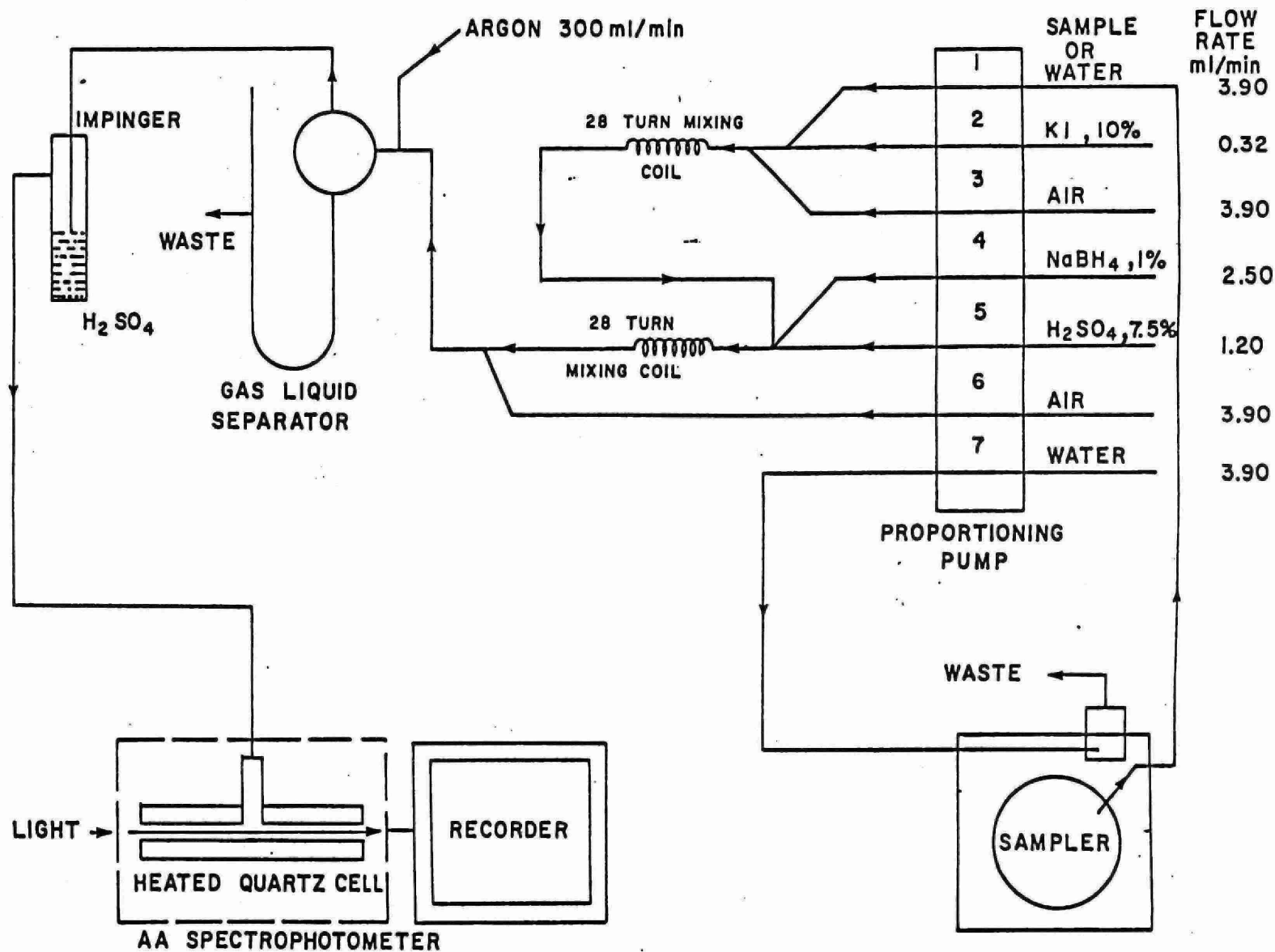


FIGURE 1 AUTOANALYSER-AAS SYSTEM DIAGRAM FOR ANTIMONY.

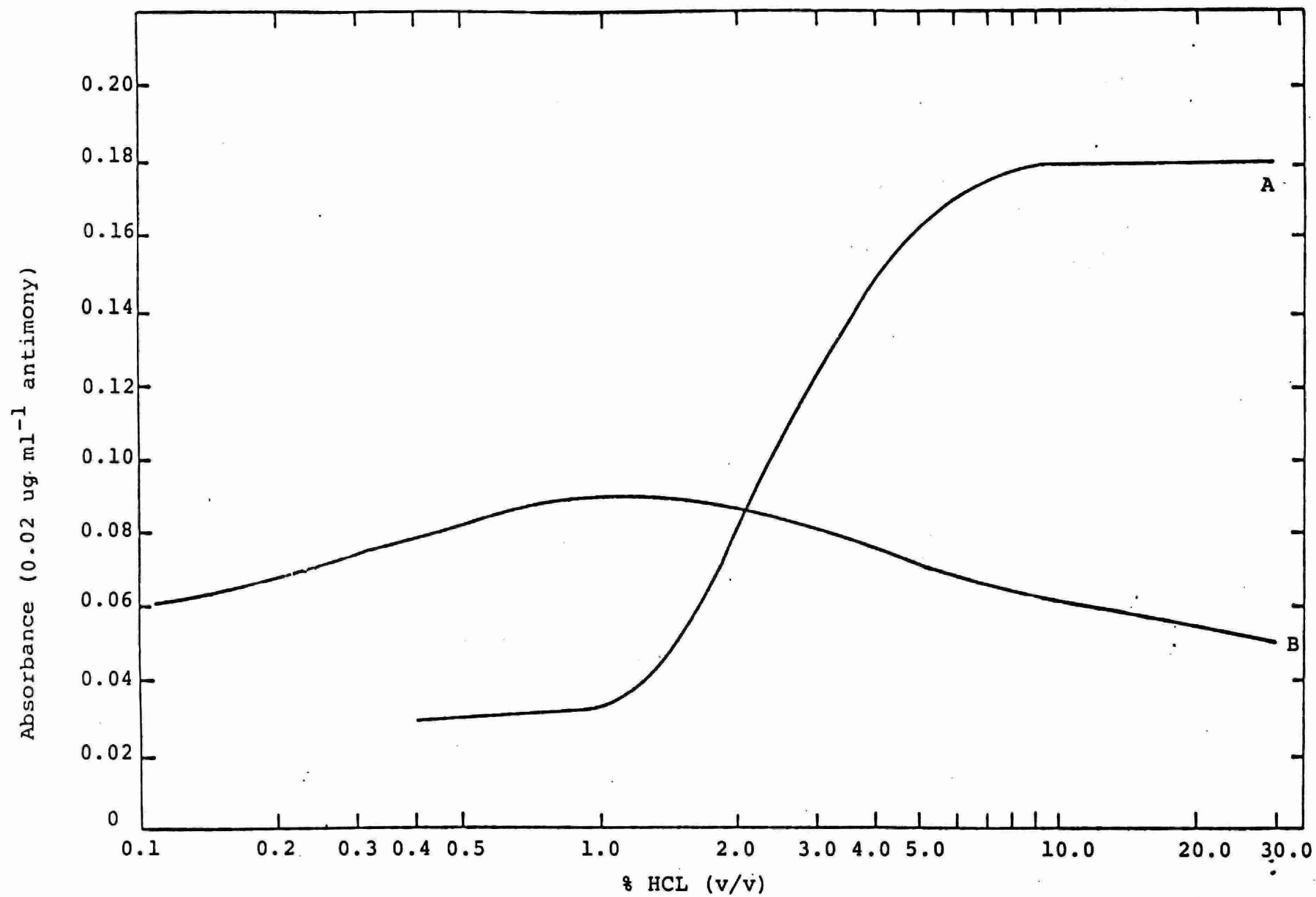


Figure 2 Effect of acid concentration on the absorbance signal of antimony (pentavalent)

A Sample acid concentration vs absorbance in presence of KI  
 B " " " " " " absence " "

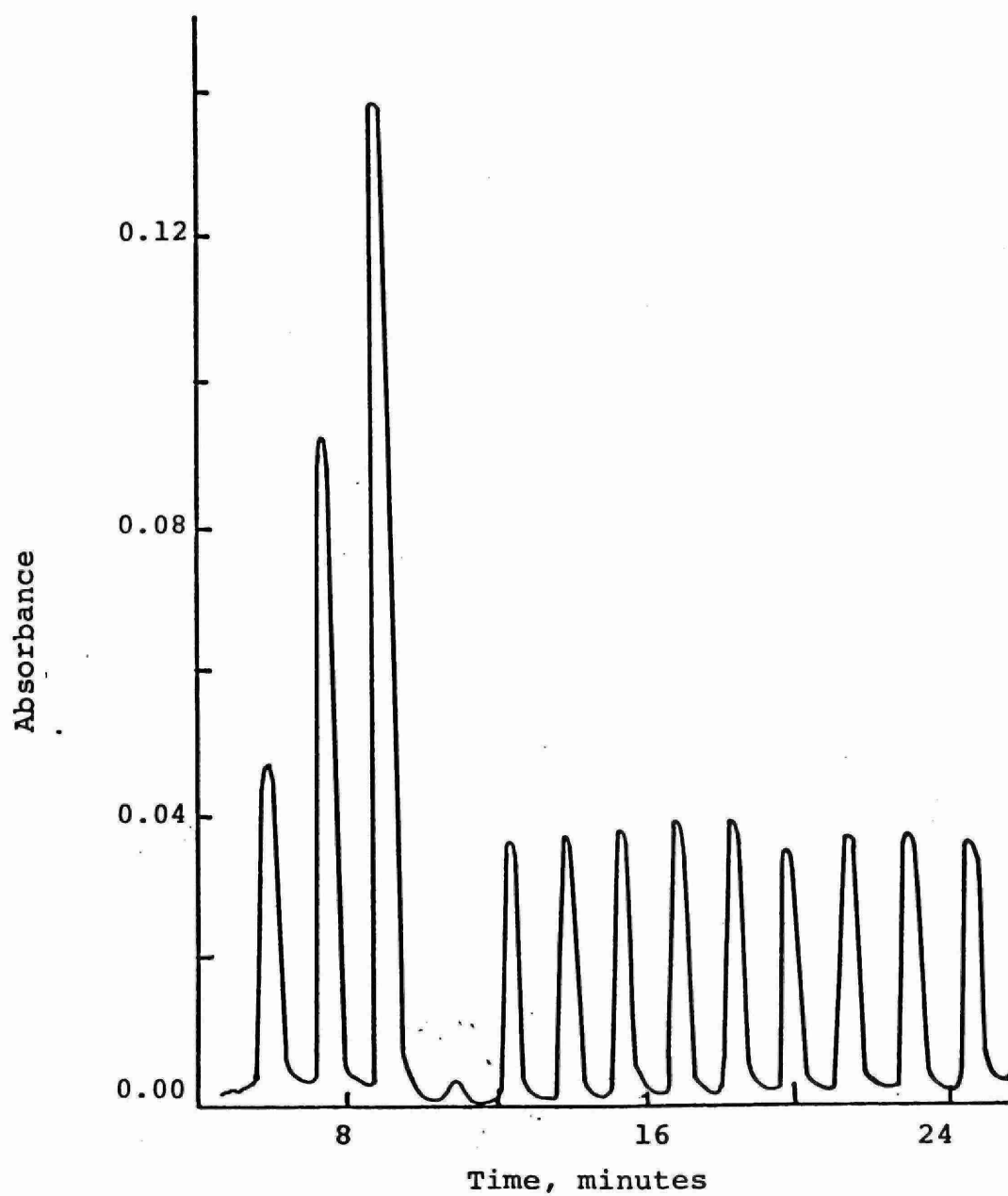


Figure 3. Typical signal peaks for antimony: 5, 10 and 15 ng/ml; blank and 10 replicates.



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